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| CHARGE NUMBER: | 6908 |
| PROGRAM TITLE: | CHEMICAL STUDIES OF CONDENSATE |
| PROJECT LEADER: | A. H. Warfield |
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PAH (R. Levins)

PAH III fractions and the strippings from the Florisil PR column used to generate the PAH III fractions from eight model cigarettes have been analyzed by GC with FID and NPD, and also submitted to Project 6906 for S/M assay (TA100 + S9). The strippings contain relatively polar compounds, possibly N-PAHs. Specific activities of the strippings were generally somewhat lower than those of the PAH-IIIs. Analysis of the GC data is in progress.

NITROSAMINE ANALYSIS (C. McKay, J. Millham, A. Warfield)

Efforts have been directed at improving the methodology employed in the analysis of volatile and nonvolatile nitrosamines. It was demonstrated that the $^{14}C$ labeled dimethylnitrosamine (NDMA) added to determine recovery does not interfere with NDMA quantitation. The steam bath used in sample concentration was replaced with a more precisely controlled water bath. Gas chromatographic conditions for nonvolatile nitrosamine determination were changed to diminish analysis time, and the chromatograph modified to enhance injection reproducibility. Studies were conducted to establish the optimum pyrolyzer temperature on the Model 610 TEA. N-nitrosodibutylamine was found to be a satisfactory internal standard for the analysis of volatile nitrosamines. Evaluation of the nonvolatile nitrosamine standards is now in progress.

BASE FRACTION ANALYSES (S. Tafur, R. Kinser, M. Zimmermann, C. Ellis)

Sephadex-LH20 fraction #7 from X6D3IM bases was separated by preparative LC. All the fractions generated were active in the S/M assay (Charge No. 6906). Chemical profiling is being used to guide isolation attempts. GC/MS profiles of seven fractions were generated. Harman and norharman are prominent components distributed throughout the fractions. Other compounds observed are in the molecular weight range 180-250 u and have mass spectra characteristic of aromaticity.

The nicotyrine level in the base fraction from LTF-5A + nicotine CSC was found to be 9.5%.

Levels of harman (H), norharman (NH), and 2-amino-α-carboline (2AC) were determined (by HPLC) in base fractions from 2R1 mainstream (MS) and sidestream (SS) smoke. Collection methods used were impaction trap (IT) (MS and SS), acid

filled impaction trap (AIT) (MS and SS), and electrostatic precipitation (EPPT) (SS only). The SS levels of 2AC, H, and NH were less than MS levels for IT and AIT samples, but EPPT SS contained 2AC, H, and NH at levels comparable to those of AIT and IT MS.

TOXICITY OF PUPAL CELL CASING (S. Tafur, R. Levins, R. Kinser, M. Zimmermann)

Collaborative studies with Dr. D. Faustini to isolate the component(s) of pupal cell casings (PCC) responsible for the observed mortality of cigarette beetles (CB) fed mixtures of PCC and bright (Br) tobacco continued. Hexane extracts of PCC and Br loaded on Br at 5% and 1% levels resulted in CB mortality, but the Br hexane extract was of greater toxicity. GC and GC/MS analyses of the hexane extracts indicated that nicotine, neophytadiene, and waxes were the primary components. A $CH_2Cl_2$:1.0 N HCl partition resulted in nicotine levels in both hexane extracts of <0.1%; the denicotinized extracts were not toxic suggesting that the toxicity observed for the hexane extract was due to nicotine. The PCC residue after hexane extraction was toxic, suggesting that the active agent was not extracted.

Insufficient amounts of material for feeding studies were obtained from $CH_2Cl_2$ extraction of the residue. Methanol extraction yielded a hygroscopic material which was loaded on Br tobacco at 15% levels for testing. Residues from hexane and methanol extracted PCC and Br tobacco are being tested at 30% loadings.

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